CHEMICAL ANALYSIS ~~TESTING~~ OF CEMENT

1.   SCOPE: This method is a modification of ASTM C-114 for the chemical testing of cement by x-ray spectroscopy.

2.   APPARATUS –MATERIALS:

   2.1.   Philips MagiX PRO Wavelength Dispersive x-ray fluorescence spectrometer.

   2.2.   SuperQ software

   2.3.   Philips Perl'X 3 fused bead machine

   2.4.   Platinum dish and crucible set

   2.5.   Spex 8000 mixer/mill

   2.6.   Lithium Bromide (LiBr): 10% solution non-wetting agent

   2.7.   100% Lithium Tetraborate ($Li_2B_4O_7$) flux

3.   PROCEDURE:

   3.1.   Prepare porcelain crucibles by igniting at 950°C to a constant weight. Cool and store in a desiccator to avoid absorption of moisture.

   3.2.   Weigh 1.0 gram of cement sample accurately to 0.0001 grams into a prepared porcelain crucible. Ignite sample to a constant weight in a muffle furnace at 950°C and cool in a desiccator.

   3.3.   Calculate the loss on ignition (LOI) using the following formula:

   LOI = (A/B) x 100

   where: A = weight of sample after ignition;
            B = weight of original sample.

   3.4.   Place at least 1.0 gram of cement sample from LOI in the spex 8000 mixer/mill and grind until a fine powder is obtained. This takes approximately 30 seconds.

   3.5.   Weigh accurately to 0.0001 grams 6.0 grams of flux directly into platinum crucible. Then weigh accurately to 0.0001 grams 0.6 grams of cement sample from the Spex 8000 mixer/mill directly into the platinum crucible. Add 3 drops of LiBr solution. Place the platinum crucible and dish in the Perl'X 3 machine and select to run program 9* for all

types of cement. This takes approximately 15 minutes. Program run is dependent on sample type.

    3.6.   Enter names of samples and LOI information in measure sample screen on the measure and analyze program.

    3.7   Place sample in a 27mm steel cup. Then place in x-ray instrument and prepare to run cement application on the measure sample screen. Click on measure at the bottom of the screen. This may take a few minutes. The application chosen is dependent upon the sample type.

4.   QUANTIFICATION: Program quantifies data by using a least squares program. Similar samples with known chemical make-ups are used as standards in the quantification technique. As many standards as possible are used for best quantification. The results are reported as oxides in weight percents.

5.   REPORT:

    5.1.   % LOI

    5.2.   % Insolubles

    5.3.   % SO_3

    5.4.   % Al_2O_3

    5.5.   % Fe_2O_3

    5.6.   % C_3A $(2.650 \times \% Al_2O_3) - (1.692 \times \% Fe_2O_3)$

    5.7.   % MgO

    5.8.   % Na_2O

    5.9.   % K_2O

    5.10.   % CaO

    5.11.   % SiO_2

* NOTE: Program 9 includes the following: One oxidation for 2 minutes, temperature 1100°C, power of generator 77, agitation angle 25, and agitation speed 10. One fusion for 6 minutes, temperature 1100°C, power of generator 77, agitation angle 50 and agitation speed 15. Then there is a pause before casting for 10 seconds at a temperature of 1100°C. Casting lasts 2 minutes, temperature 1100°C, casting angle 123, casting speed 10 and time for solidification is 30 seconds. Lastly, there is natural cooling for 1 minute and forced air cooling for 3 minutes at a flow rate of 40. The setting of the dish height dial is 40/12 and this depends on the size of the platinum dish used.

1.	SCOPE: This method is a modification of ASTM C-114 for the chemical testing of cement by X-ray spectroscopy.

2.	APPARATUS and REAGENTS:

	2.1.	Kevex – Analyst 771 Energy Dispersive X-Ray Fluorescence Spectrometer.

	2.2.	WinXRF software

	2.3.	Carver hydraulic press

	2.4.	Hardened Steel 31mm die set

	2.5.	Spex 8000 mixer/mill

	2.6.	Polyvinyl Alcohol (PVA)

3.	PROCEDURE:

	3.1.	Weigh 6 grams of cement sample accurately to 0.0001 grams. Weigh accurately to 0.0001 grams 0.9 grams of polyvinyl alcohol. Calculate the added/sample ratio. Calculation:

		Weight polyvinyl alcohol/weight of cement = Added/Sample Ratio

	3.2.	Place both components in the Spec 8000 mixer/mill, and grind until a fine power is obtained and/or sample is completely uniform. This takes approximately 30 seconds.

	3.3.	Place the homogeneous sample in the die. Press at 33,000 lbs and allow pressure to release very slowly. This make take up to 45 minutes.

	3.4.	Enter names of samples and Added/Sample ratio information by running NAMES program in the cement library.

	3.5.	Place sample in machine and prepare to run program CEMENT.

	3.6.	Condition Codes: Program will run through a series of condition codes. The condition code acquires a spectrum, subtracts escape peaks, subtracts the background, and deconvolutes for indicated elements. The peak intensities are saved. Condition code values are show in Table 3.1.

		3.6.1.	Code 1: Deconvolutes spectra for Al, Si, S, and P.

		3.6.2.	Code 2: Deconvolutes spectra for K, Ti, Mn, Fe, and Ca.

		3.6.3.	Condition Code 3: Deconvolutes spectra for Mg and Na.

	3.7.	Quantification: Program quantifies data by using a Least Squares Program. Similar

samples with known chemical makeups are used as standards in quantification technique. As many standards as possible are used for the best quantification. The results are reported as oxides.

TABLE 3.1 CONDITIONS

Condition Code	kV	mA	Secondary Target	Acquire Time (Sec)	Spectra Size
1	5.0	.10	Direct	400	1024
2	20.0	1.45	GE	400	2048
3	25.0	3.00	AL	400	1024

All conditions are set: Gain = 40, Shaping Index = 32, eV/Channel = 10

APPROVED   _____

Director
DIVISION OF MATERIALS

DATE _____1/6/03_____

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